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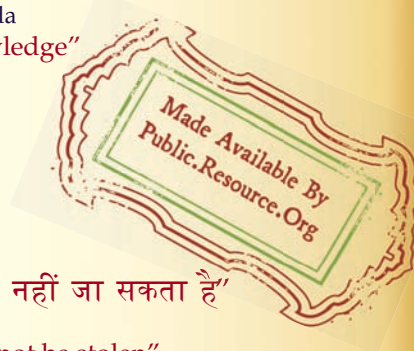
IS 14742 (1999): Textile Auxiliaries - Silicone Emulsion
[TXD 7: Textile Sizing and Finishing Materials]



“ज्ञान से एक नये भारत का निर्माण”

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“Invent a New India Using Knowledge”



“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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भारतीय मानक

वस्त्रादि सहायक — सिलिकॉन इमल्शन — विशिष्टि

Indian Standard

TEXTILE AUXILIARIES — SILICONE EMULSION —
SPECIFICATION

ICS 59.080.01; 71.100.40

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Textile Sizing and Finishing Materials Sectional Committee had been approved by the Textile Division Council.

Silicone emulsions are extensively used in the textile industry for textile finishing as softeners, water repellent and water proofing, antislip coating and fine coating of textiles. The use of silicone emulsion helps considerable saving in processing cost and gives the final product an excellent finish. For textile finishing two different types of silicone emulsions are used. These are:

- i) A reactive polymethyl hydrogen siloxane, and
- ii) A non-reactive polydimethyl siloxane.

The reactive polymethyl hydrogen emulsions are used along with the softener or plasticizer. The non-reactive polydimethyl emulsions are used in conjunction with titanium or zirconium compound for water repellency.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be same as that of the specified value in this standard.

Indian Standard

TEXTILE AUXILIARIES — SILICONE EMULSION — SPECIFICATION

1 SCOPE

This standard prescribes the requirements for silicone emulsions of polydimethyl siloxane and polymethyl hydrogen siloxane used in textile industry as finishing agents.

2 REFERENCES

The following standards contain provisions which through reference in this text, constitute provision of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

<i>IS No.</i>	<i>Title</i>
1070 : 1992	Water for general laboratory use (third revision)
1390 : 1983	Methods for determination of pH value of aqueous extracts of textile materials (first revision)

3 REQUIREMENTS

Silicone emulsion shall meet the requirements as given in Table 1.

4 SAMPLING AND CRITERIA FOR CONFORMITY

4.1 Sampling

4.1.1 Lot

All the containers of one definite type and quality of silicone emulsion and of the same composition delivered to a buyer against one despatch note shall constitute a lot.

4.1.2 Unless otherwise agreed to between the buyer and the seller, the number of containers to be selected from each lot at random shall be as given below:

<i>Lot Size</i>	<i>Sample Size</i>
2 to 15	2
16 „ 25	3
26 „ 50	4
51 „ 100	5
101 „ 150	6
151 „ 300	7
301 and above	8

4.1.3 Stir the contents of each container, as selected in 4.1.2, thoroughly for not less than 5 minutes with a clean stick before sampling. The stirring may be prolonged particularly if emulsion is not homogenous. After ensuring that the contents in the container are completely homogenized, draw a sample of about 100 g in a clean bottle which should be stoppered immediately after filling. This shall constitute the test sample. The test sample should be stirred again with a clean glass rod just before weighing in various test methods given in this standard.

4.2 Criteria for Conformity

4.2.1 The lot shall be considered conforming to the requirements of this standard if the composite sample meets the various requirements specified under 3.

5 MARKING

5.1 Each package shall be marked with the following information:

- a) Net mass of the emulsion;
- b) Name of the material, namely, silicone emulsion;
- c) Solid content of the material;
- d) Indication of the source of manufacture; and
- e) Month and year of manufacture.

5.2 BIS Certification Marking

Each package may also be marked with the Standard Mark.

5.2.1 The use of the Standard Mark is governed by the provisions of *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for use of Standard Mark may be granted to manufacturers or producers may be obtained from Bureau of Indian Standards.

6 PACKING

The material shall be packed in adequately waterproof packages providing total protection from moisture, dirt, dust, etc, as detailed in the contract or order.

Table 1 Requirements of Silicone Emulsion
(Clause 3)

Sl No.	Characteristic	Requirement for		Method of Test
		Polydimethyl Siloxane	Polymethyl Hydrogen Siloxanes	
(1)	(2)	(3)	(4)	(5)
i)	Physical appearance	White emulsion	White emulsion	Visual examination
ii)	pH	5-6	5-6	pH meter
iii)	Non-volatile matter, percent	30 ± 2	30 ± 2	Annex A
iv)	Oil content, percent	25 ± 2	25 ± 2	Annex B
v)	Silica content, as SiO ₂ , percent	20 ± 1	25 ± 1	Annex C
vi)	Active matter, percent (based on silica), percent	25 ± 2	25 ± 2	Annex D

ANNEX A

[Table 1, Sl No. (iii)]

ESTIMATION OF NON-VOLATILE MATTER

A-1 PRINCIPLE

The emulsion is dried in an oven to a constant mass and non-volatiles (solid content) is calculated by expressing dried residue (solids) as percentage of the original mass of the sample under test.

A-2 APPARATUS

- Petri dish,
- Air drying oven capable of operating at 105 ± 3°C,
- Desiccator (20 cm dia) with calcium chloride as desiccant, and
- A weighing balance with accuracy up to 1 mg.

A-3 OVEN DRYING METHOD

Weigh 2 to 3 g of silicone emulsion in a clean, dry and tared petri dish and dry in an air oven at a temperature of 105 ± 3°C for a minimum period of 4 hours, but preferably to constant mass (*see Note*). Calculate the non-volatile matter as follows:

Total non-volatile

$$\text{Matter, percent} = \frac{\text{Mass in g of dried residue}}{\text{Mass in g of sample taken}} \times 100$$

NOTE—Weigh the container along with dried residue after 4 hours and then at an interval of 1 hour. If the difference in mass is less than 0.20 percent, then the same should be taken as constant mass.

ANNEX B

[Table 1, Sl No. (iv)]

ESTIMATION OF SILICONE OIL CONTENT

B-1 PRINCIPLE

The cage like structure formed in the emulsion by bridging of oil and water with the help of emulsifiers, is broken down by 1, 4 dioxane because it has more affinity with water than silicone due to its solvent polarity and density.

B-2 PROCEDURE

To a measured quantity of silicone emulsion, say 30 ml, add 30 ml of 1, 4 dioxane in graduated, 100 ml cylinder. Shake well and allow it to stand

for four hours. Emulsion will break into three crystal clear layers as follows:

- Water and 1, 4 dioxane* — clear, colourless liquid at the bottom of cylinder;
- Emulsifiers* — a thin layer between the water and oil layer; and
- A crystal clear layer of oil on top (to be tested subsequently for purity).

Read off the quantity of top layer and relate this to the quantity of silicone emulsion tested. For example in a 100 ml sample, a 30 ml top layer indicates a 30 percent silicone oil content.

ANNEX C

[Table 1, Sl No. (v)]

ESTIMATION OF SILICA CONTENT

C-1 PRINCIPLE

A weighed quantity of silicone emulsion is reduced to silica by destroying the organic matter wet ashing procedure. The precipitated silica is filtered, washed, dried and ashed and expressed as percent of the sample taken.

C-2 APPARATUS

- Analytical balance with accuracy up to 1 mg,
- Air drying oven, capable of operating at $105 \pm 3^\circ\text{C}$,
- Hot plate,
- Desiccator (20 cm dia) with calcium chloride as desiccant,
- Borosilicate glass beakers, watch glasses, funnels, platinum or silica crucibles, and
- Whatman filter paper No. 40 or its equivalent.

C-3 REAGENTS

The acids used for wet ashing should be of AR grade of highest purity, namely, sulphuric acid, nitric acid, perchloric acid and hydrochloric acid.

C-4 PROCEDURE

- Weigh accurately about 0.5 to 1 g of the sample and transfer it to clean dry 500 ml glass beaker covered with a watch glass.
- Add 30 ml of sulphuric acid, followed by gradual addition of nitric acid (this volume can vary and may be as much as 30 ml).

- Apply heat gradually on hot plate in a fume cupboard and digest with further addition of nitric acid (*see Note*), if necessary, until the liquid is clear.
- Evaporate down 20 to 25 ml. Cool to room temperature, dilute cautiously with 100-150 ml of distilled water and add 1-2 ml of hydrochloric acid.
- Boil the contents to remove nitric acid.
- Filter the solution while hot on a Whatman filter paper No. 40 or its equivalent. Make sure that the entire precipitate is removed from the beaker as the silica is very adherent. Wash the precipitate several times with hot water until free from acid (check the filtrate with methyl orange indicator and there will be no red colour if free from acid).
- Dry the precipitate with the funnel in the oven maintained at $105 \pm 3^\circ\text{C}$ for 1 h. Transfer the filter paper with the precipitate to a weighed platinum or silica crucible. Burn off the filter paper on a slow flame and transfer the crucible to a muffle furnace and ash at 800°C for 30 minutes.
- Cool the crucible with the ash in a desiccator to room temperature and weigh.

Silica content as

$$\text{SiO}_2 \text{ percent} = \frac{\text{Weight of the ash}}{\text{Weight of sample}} \times 100$$

NOTE — Cool the beaker and its contents to a moderate temperature before adding nitric acids at regular intervals. Adding nitric acid to hot contents in the beaker will cause spurting.

ANNEX D

[Table 1, Sl No. (vi)]

ESTIMATION OF ACTIVE MATTER, PERCENT

D-1 The active matter may be calculated from silica content obtained above as follows:

- Silicone content, percent = Silica as SiO_2 (polydimethyl siloxane type) percent $\times 1.241$

- Silica content, percent = Silica as SiO_2 (polymethyl hydrogen siloxane type) percent $\times 1.006$

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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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